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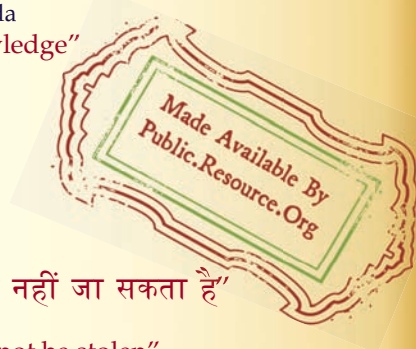
IS 6507 (1991): Sodium chromate, technical [CHD 1:
Inorganic Chemicals]



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भारतीय मानक
सोडियम क्रोमेट, टेट्राहाइड्रेट — विशिष्टि
(दूसरा पुनरीक्षण)
Indian Standard
SODIUM CHROMATE, TETRAHYDRATE —
SPECIFICATION
(*Second Revision*)

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BUREAU OF INDIAN STANDARDS
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FOREWORD

This Indian Standard (Second Revision) was adopted by the Bureau of Indian Standards, after the draft finalized by the General Inorganic Chemicals Sectional Committee had been approved by the Chemical Division Council.

Sodium chromate is used in the manufacture of pigments and as raw material for other chromium chemicals. It is also used for corrosion inhibition.

This standard was first published in 1972 and revised in 1982 upgrading the purity to 98.5 percent from 98 percent as existing in the original standard.

In this revision, two grades of sodium chromate, tetrahydrate have been prescribed, namely, pigment grade and chemical grade.

For the purpose of deciding whether a particular requirement of this standard is complied with, the final value, observed or calculated, expressing the result of a test or analysis, shall be rounded off in accordance with IS 2 : 1960 'Rules for rounding off numerical values (*revised*)'. The number of significant places retained in the rounded off value should be the same as that of the specified value in this standard.

Indian Standard

SODIUM CHROMATE, TETRAHYDRATE — SPECIFICATION

(Second Revision)

1 SCOPE

This standard prescribes the requirements and the methods of sampling and test for sodium chromate, tetrahydrate.

2 REFERENCES

The Indian Standards listed below are necessary adjuncts to this standard.

IS No.	Title
264 : 1976	Nitric acid (<i>second revision</i>)
265 : 1987	Hydrochloric acid (<i>third revision</i>)
323 : 1959	Rectified spirit (<i>revised</i>)
695 : 1986	Acetic acid (<i>third revision</i>)
1070 : 1977	Water for general laboratory use (<i>second revision</i>)
4905 : 1968	Methods for random sampling

3 GRADES

The material shall be of following two grades:

- Grade 1 — Pigment grade, and
- Grade 2 — Chemical grade.

4 REQUIREMENTS

4.1 Description

Sodium chromate, tetrahydrate shall be in the form of yellow crystals, deliquescent and soluble in water.

4.2 The material shall comply with the requirements laid down in Table 1 when tested according to the methods prescribed in Annex A. Reference to the relevant clauses of Annex A is given in col 5 of the table.

5 PACKING AND MARKING

5.1 Packing

The material shall be packed in polyethylene lined paper or hessian bags or mild steel drums.

Table 1 Requirement for Sodium Chromate, Technical
(Clause 4.2)

Sl No.	Characteristic	Requirement		Method of Test (Ref to Cl. No. in Annex A)
		Grade 1	Grade 2	
(1)	(2)	(3)	(4)	(5)
i)	Total moisture (free moisture and combined water), percent by mass, <i>Max</i>	31.5	33.0	A-3
ii)	Sodium chromate (as Na_2CrO_4), percent by mass, <i>Min</i>	98.5	97.0	A-4
iii)	Sulphate (as Na_2SO_4), percent by mass, <i>Max</i>	1.00	2.5	A-5
iv)	Chlorides (as NaCl), percent by mass, <i>Max</i>	0.50	0.50	A-6

NOTE — The requirements for Sl No. (ii) to (iv) are on dry basis.

5.2 Marking

The containers shall be legibly and indelibly marked with the following information:

- a) Name and grade of the material;
- b) Indication of the source of manufacture;
- c) Net mass of the contents;
- d) Month and year of manufacture; and
- e) Batch number.

5.2.1 The containers shall be marked with the following caution note:

'TOXIC — HANDLE WITH CARE'

6 SAMPLING

The method of drawing representative samples of the material, number of tests to be performed and the criteria for conformity of the material to the requirements of this specification shall be as prescribed in Annex B.

ANNEX A

(Clause 4.2 and Table 1)

METHODS OF TEST FOR SODIUM CHROMATE, TECHNICAL

A-1 QUALITY OF REAGENTS

A-1.1 Unless otherwise specified, pure chemicals and distilled water (see IS 1070 : 1977) shall be used in tests.

NOTE — 'Pure chemicals' shall mean chemicals that do not contain impurities which affect the results of analysis.

A-2 PREPARATION OF SAMPLE SOLUTION

A-2.1 Procedure

Weigh accurately about 25 g of the material in either silica or porcelain dish and dry to constant mass over either a small flame using wire gauze or in an oven at $125^{\circ} \pm 5^{\circ}\text{C}$. The flame shall be sufficiently small so as not to allow the material to get spurted while drying. Weigh and note the exact mass of the dried sample and transfer completely to a 500 ml volumetric flask. Add water and make up the volume to 500 ml mark. This prepared solution shall be used in tests prescribed in A-4 to A-6.

A-3 TOTAL MOISTURE (FREE MOISTURE AND COMBINED WATER)

A-3.1 Procedure

Weigh accurately about 5 g of the material in either silica or porcelain dish of about 75 ml capacity. Dry the contents to constant mass over a small flame using wire gauze or in an oven at $125^{\circ}\text{C} \pm 5^{\circ}\text{C}$. The flame shall be sufficiently small so as not to allow the material to get spurted while drying. It takes about 45 minutes at dry completely over a flame and about 6 hours in an oven.

A-3.2 Calculation

Total moisture, percent by mass

$$= \frac{(M - M_1)}{M} \times 100$$

where

M = mass in g of the material taken for the test, and

M_1 = mass in g of the material after drying.

A-4 SODIUM CHROMATE

A-4.1 Reagents

A-4.1.1 *Standard Potassium Permanganate Solution* — 0.05 N.

A-4.1.2 *Ferrous Ammonium Sulphate Solution* — approximately 0.1 N.

A-4.1.3 *Dilute Sulphuric Acid* — approximately 2 N.

A-4.2 Procedure

Pipette out 50 ml of the prepared sample solution (see A-2) in a 500 ml volumetric flask and make up the volume with water. Take out 25 ml of the solution in a conical flask. Add 25 ml of dilute sulphuric acid and 25 ml of ferrous ammonium sulphate solution. Titrate the excess of ferrous ammonium sulphate solution against standard potassium permanganate solution. Conduct a blank titration with 25 ml of ferrous ammonium sulphate solution against standard potassium permanganate solution.

A-4.3 Calculation

Sodium chromate (as Na_2CrO_4),

$$\text{percent by mass} = \frac{(V_1 - V_2) \times 2 \times N \times 54}{M}$$

where

V_1 = volume in ml of potassium permanganate solution used in the blank titration,

V_2 = volume in ml of potassium permanganate solution used in the titration,

N = normality of standard potassium permanganate solution, and

M = mass in g of the material present in the aliquot of the solution.

A-5 SULPHATES

A-5.1 Reagents

A-5.1.1 *Concentrated Hydrochloric Acid* — see IS : 265 : 1987.

A-5.1.2 *Rectified Spirit* — see IS 323 : 1959.

A-5.1.3 *Glacial Acetic Acid* — see IS 695 : 1986.

A-5.1.4 *Barium Chloride Solution* — approximately 12 percent (m/v).

A-5.1.5 *Dilute Hydrochloric Acid* — approximately one percent (v/v).

A-5.2 Procedure

Concentrate 200 ml of prepared sample solution (see A-2) to about 50 ml. Add 10 ml of concentrated hydrochloric acid, 15 ml of rectified spirit and 35 ml of glacial acetic acid. Boil for 15 minutes. Dilute to about 200 ml. To the hot solution add 20 ml of hot barium chloride solution slowly with constant stirring, to precipitate the sulphate. Continue boiling

for 15 minutes and allow it to stand for 4 hours. Filter the solution containing barium sulphate through a tared sintered glass crucible No. G 4 or on a Gooch crucible or a filter paper (Whatman No. 42 or equivalent). Wash with dilute hydrochloric acid till the filtrate is free from chromium salts and then with water till it is free from chlorides.

A-5.2.1 If the filtration has been done through a sintered glass crucible, dry the precipitate at $100 \pm 5^\circ\text{C}$. In case of Gooch crucible, dry the precipitate in an oven at a temperature less than 250°C . When the filtration is carried out on a filter paper, ignite the precipitate over a burner or in the muffle furnace at 600° to 700°C for half an hour. After this, cool and add concentrated nitric acid (2 drops) and gently heat to drive off acid fumes. Then ignite again till constant mass is obtained.

A-5.3 Calculation

$$\text{Sulphates (as Na}_2\text{SO}_4\text{), percent by mass} = \frac{60.86 \times M_1}{M}$$

where

M_1 = mass in g of the precipitate, and

M = mass in g of the sample present in the aliquot.

A-6 CHLORIDES

A-6.1 Reagents

A-6.1.1 Concentrated Nitric Acid — see IS 264 : 1976.

A-6.1.2 Silver Nitrate Solution — approximately 5 percent (m/v).

A-6.1.3 Dilute Nitric Acid — approximately 4 N.

A-6.2 Procedure

Take 200 ml of prepared sample solution (see A-2). Filter, if any insoluble matter is present. Add 20 ml of concentrated nitric acid and heat the solution to about 50°C . Add to the hot solution sufficient volume of silver nitrate solution to precipitate chlorides completely. Protect the silver chloride precipitate from light by wrapping black paper around the container. Cool the solution and filter through a tared sintered glass crucible No. G4. Wash the precipitate first with dilute nitric acid and dissolve the adhering silver chromate, and then with cold water. Dry the crucible and its contents to constant mass at $130 \pm 2^\circ\text{C}$.

A-6.3 Calculation

$$\text{Chlorides (as NaCl), percent by mass} = \frac{40.78 \times M_1}{M}$$

where

M_1 = mass in g of silver chloride precipitate, and

M = mass in g the sample present in the aliquot.

ANNEX B

(Clause 6)

SAMPLING OF SODIUM CHROMATE, TECHNICAL

B-1 GENERAL REQUIREMENTS OF SAMPLING

B-1.0 In drawing, preparing, storing and handling samples, the following precautions shall be observed.

B-1.1 Samples shall not be taken out in an exposed place.

B-1.2 The sampling instruments shall be clean and dry.

B-1.3 Precautions shall be taken to protect the samples, the material being sampled, the sampling instrument and the containers for samples from adventitious contamination.

B-1.4 To draw a representative sample, the contents of each container selected for sampling shall be mixed thoroughly by suitable means.

B-1.5 The samples shall be placed in suitable, clean, dry, and air-tight glass or other suitable containers on which the material has no action.

B-1.6 The sample containers shall be of such a size that they are almost but not completely filled with the sample.

B-1.7 Each sample container shall be sealed air-tight after filling, and marked with full details of sampling, the year of manufacture and other important particulars of the consignment.

B-2 SCALE OF SAMPLING

B-2.1 Lot

All the containers in a single consignment of the material drawn from a single batch of manufacture shall constitute a lot. If a consignment is declared or known to consist of different batches of manufacture, the containers belonging to the same batch shall be grouped together and each such group shall constitute a separate lot.

B-2.1.1 Samples shall be tested from each lot separately for ascertaining the conformity of the material to the requirements of the specification.

B-2.2 The number of containers (n) to be chosen from a lot shall depend on the size of the lot (N) and shall be in accordance with Table 2.

Table 2 Number of Containers to be Selected for Sampling
(Clause B-2.2)

Lot Size	Number of Containers To be Selected
N	n
(1)	(2)
3 to 50	3
51 to 200	4
201 to 400	5
401 to 650	6
651 and above	7

B-2.3 The containers to be selected for sampling shall be chosen at random from the lot and for this purpose random number tables shall be used (see IS 4905 : 1968). In case such tables are not available, the following procedure may be adopted:

Starting from any container, count them as 1, 2, 3,, up to r and so on, where r is the integral part of N/n . Every r th container thus counted shall be taken out till the requisite sample of n containers is obtained.

B-3 TEST SAMPLES AND REFEREE SAMPLES

B-3.1 Preparation of Test Samples and Referee Sample

B-3.1.1 Draw with an appropriate sampling instrument a small portion of the material from different parts of each container selected for sampling. The total quantity of the material drawn from each container shall be sufficient to make triplicate determinations for all the characteristics given under 4.

B-3.1.2 Thoroughly mix all portions of the material drawn from the same container. Out of these portions small but equal quantity shall be taken from each selected container and shall be well mixed together so as to form a composite sample weighing not less than 300 g. This composite sample shall be divided into three equal parts, one for the purchaser, the second for the supplier and the third to be used as referee sample.

B-3.1.3 The remaining portion of the material from each container (after a small quantity needed for the formation of composite sample has been taken out) shall be divided into three equal parts, each part weighing not less than 10 g. These parts shall be immediately transferred to thoroughly dried bottles which are

then sealed air-tight with stoppers and labelled with all the particulars of sampling given in B-1.7. The material in each such sealed bottle shall constitute an individual test sample. These individual samples shall be separated into three identical sets of samples in such a way that each set has an individual test samples representing each container selected. One of these three sets shall be for the purchaser, the second for the supplier and third shall be used as referee sample.

B-3.2 The referee sample consisting of the composite sample (see B-3.1.2) and a set of individual sample (B-3.1) shall be marked for this purpose and shall bear the seals of the purchaser and the supplier. It shall be kept at a place agreed to between the purchaser and the supplier and shall be used in case of dispute between the two.

B-4 NUMBER OF TESTS

B-4.1 Tests for the determination of sodium chromate shall be conducted on each of the individual samples constituting the set of test sample.

B-4.2 Tests for the remaining characteristics shall be conducted on the composite sample.

B-5 CRITERIA FOR CONFORMITY

B-5.1 For Individual Samples

The mean and the range of the test results for the determination of sodium chromate shall be calculated as follows:

Mean (\bar{X}) = the sum of the test results divided by the number of the test results, and

Range (R) = the difference between the maximum and the minimum values of the test results.

B-5.1.1 If ($\bar{X} - 0.6 R$) is equal to or greater than the minimum requirement of sodium chromate, the lot shall be declared to have satisfied the requirement for sodium chromate.

B-5.2 For Composite Samples

The test results on the composite sample shall meet the corresponding requirements specified in 4.

B-5.3 A lot shall be declared as conforming to this specification if it satisfies the requirements of the characteristics specified in 4.

B-5.4 If the requirements for any of the characteristics are not met, the lot shall be declared to have not satisfied the requirements of this specification.

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